570990NPS4

United States
Environmental Protection
Agency

Office of Water
Office of Pesticides and
Toxic Substances

Fall 1990

EPA National Pesticide Survey

Analytical Methods

The U.S. Environmental Protection Agency (EPA) has completed its five-year National Survey of Pesticides in Drinking Water Wells (NPS). A joint project of EPA's Office of Drinking Water (ODW) and Office of Pesticide Programs (OPP), the Survey was designed to assess the extent and severity of the presence of pesticides and nitrate in drinking water wells nationwide, and the relationship of pesticide use and ground-water vulnerability to the presence of pesticides and nitrate.

In designing the Survey, EPA selected chemicals (called Survey analytes) to test for, and identified appropriate testing methods (called Survey analytical methods) for the detection of these analytes. This fact sheet describes how EPA selected analytes, identified analytical methods, and used the analytical methods in the laboratories.

How Were Analytes Selected?

EPA tested water samples from 783 rural domestic wells and 566 community water system wells, nationwide, for the presence of 101 pesticides, 25 pesticide degradates, and nitrate (a total of 127 Survey analytes). These analytes were chosen by EPA from among approximately 600 pesticides registered for agricultural use by EPA. EPA identified those that had:

- chemical/physical properties, including water solubility, partition coefficients, field half-life, and hydrolysis half-life that indicated a potential to leach to ground water; and
- at least one million pounds or more of use nationwide in 1982.

EPA automatically selected all pesticides regulated by the Safe Drinking Water Act, plus pesticides detected in ground-water studies done prior to the NPS.

EPA selected a final list of 127 analytes based on the criteria listed above for which an analytical method existed or could be developed, plus other chemicals that could be analyzed at the same time as the selected analytes that met the listed criteria, without additional cost. The Survey Analytes Fact Sheet provides detailed information on all Survey analytes.

Determining Analytical Methods

Through extensive literature searches and consultation with scientific experts, EPA identified existing laboratory methods for most of the analytes. Because of the wide variety in procedures used in these methods and the large number of analytes to be included in the Survey, EPA needed methods that could efficiently test for multiple analytes. In the end, EPA selected two existing EPA methods (NPS Methods 7 and 9) and developed six new methods (NPS Methods 1-6). One of the new methods tests for ethylene thiourea and the other five, referred to as multi-residue methods, are each capable of detecting ten or more analytes (NPS Methods 1-5). EPA dropped one potential method (NPS Method 8). NPS Method 9 analyzes the combined presence of nitrate and nitrite and expresses the result as nitrogen (N).

A short description of the eight analytical methods, which includes the type and number of analytes, is presented in Exhibit 1.

Exhibit 1
Analytes Detectable By Method

NPS METHOD 1: Gas Chromatography with a Nitrogen-Phosphorous Detector			(46 Analytes)
Alachlor	Diphenamid	Methyl paraoxon	Simazine
Ametryn	Disulfoton*	Metolachlor	Simetryn
Atraton	Disulfoton sulfone*	Metribuzin	Stirofos
Atrazine	Disulfoton sulfoxide*	Mevinphos	Tebuthiuron
Bromacil	EPTC	Molinate	Terbacil
Butachlor	Ethoprop	Napropamide	Terbufos*
Butylate	Fenamiphos	Norflurazon	Terbutryn
Carboxin	Fenarimol	Pebulate	Triademefon
Chlorpropham	Fluridone	Prometon	Tricyclazole
Cycloate	Hexazinone	Prometryn	Vernolate
Diazinon*	MGK 264	Pronamide*	
Dichlorvos	Merphos*	Propazine	

NPS METHOD 2: Gas Chromatography with an Electron Capture Detector			(29 Analytes)
4,4-DDD	Dieldrin	Heptachlor epoxide	gamma - HCH
4,4-DDE	Endosulfan I	Hexachlorobenzene	alpha-Chlordane
4,4-DDT	Endosulfan II	Methoxychlor	gamma-Chlordane
Aldrin	Endosulfan sulfate	Propachlor	cis - Permethrin
Chlorobenzilate*	Endrin	Trifluralin	trans - Permethrin
Chloroneb	Endrin aldehyde	alpha - HCH	
Chlorothalonil	Etridiazole	beta - HCH	
DCPA	Heptachlor	delta - HCH*	

NPS METHOD 3: Gas Chromatography with an Electron Capture Detector			(17 Analytes)
2,4-D	4-Nitrophenol*	Dalapon*	Pentachlorophenol (PCP)
2,4-DB	Acifluorfen*	Dicamba	Picloram
2,4,5-TP	Bentazon	Dicamba, 5-hydroxy-	
2,4,5-T	Chloramben*	Dichlorprop	
3,5-Dichlorobenzoic acid	DCPA acid metabolites	Dinoseb	

NPS METHOD 4: High Performance Liquid Chromatography with an Ultraviolet Detector			(18 Analytes)
Atrazine, deethylated	Diuron	Metribuzin DA	Propanil
Barban	Fenamiphos sulfone	Metribuzin DADK*	Propham
Carbofuran, phenol-3-keto-	Fenamiphos sulfoxide	Metribuzin DK*	Swep
Carbofuran, phenol	Fluometuron	Neburon	
Cyanazine	Linuron	Pronamide metabolite	

NPS METHOD 5: Direct Aqueous Injection HPLC with Post-Column Derivatization			(10 Analytes)
Aldicarb	Baygon	Carbofuran, 3-hydroxy-	Oxamyl
Aldicarb sulfone	Carbaryl	Methiocarb	
Aldicarb sulfoxide	Carbofuran	Methomyl	

NPS METHOD 6: Gas Chromatography with a Nitrogen-Phosphorous Detector	(1 Analyte)
Ethylene thiourea (ETU)	

NPS METHOD 7: Microextraction and Gas Chromatography			(5 Analytes)
Ethylene dibromide (EDB)	1,2 - dichloropropane**	trans - 1,3 -	
Dibromochloropropane (DBCP)	cis - 1,3 - dichloropropene**	dichloropropene**	

NPS METHOD 9: Automated Cadmium Reduction and Colorimetric Detection	(1 Analyte)
Nitrate and nitrite measured as nitrogen (N)	

* Qualitative only.
** Method 8 dropped. Analytes previously included in Method 8 also detectable by Method 7.

In the Laboratory

EPA contracted with five laboratories to perform one or more of the NPS analytical methods. Two EPA laboratories managed the contracts with the contract laboratories, provided technical support, reviewed the data from the laboratories and carried out referee analyses. A third EPA laboratory provided laboratory analysis support. In addition to the quality assurance procedures carried out by the contract laboratories, the EPA laboratories analyzed duplicate samples for approximately 10 percent of the field samples collected, depending on the analytical method. In this way, EPA ensured the accuracy of results.

Rigorous quality control (QC) procedures were followed throughout the Survey. All positive detections of analytes using a gas chromatograph (GC) column (NPS Methods #1,2,3,6 and 7) were confirmed by reanalyzing samples using a gas chromatograph/mass spectrometer (GC/MS) or a different quantitative and qualitative high performance liquid chromatographic (HPLC) column (NPS Methods 4 and 5). Some GC/MS confirmations were performed by the contract laboratories; however, the majority were performed by the EPA laboratories.

The manner in which a positive detection was reported was based on the minimum quantification limit (MQL) for the analyte. The MQL is a measure of the accuracy of the analytical instrumentation and methods used to detect the analyte and below which the detected concentration is not considered reliable. Detections between one-half the MQL and the MQL indicate that an analyte was present but were reported without a concentration. A concentration could be determined for 112 of the analytes above the reporting limit. The remaining 15 were difficult to quantify or to determine a concentration level with any reliability. The Agency chose to go ahead and look for the presence of these analytes and only report them as "positive detections."

The laboratories performed a variety of QC procedures to eliminate, as much as possible, the occurrence of false negative or false positive results. These procedures included the use of laboratory and instrument control standards, analysis of method blanks (a portion of reagent water analyzed as if it were a water sample) and analysis of shipping blanks (reagent water transferred to a sampling bottle, shipped to the field, and returned to the laboratory with the samples). QC analyses enabled EPA to identify any problems with the laboratory methods or instrumentation that might affect the accuracy and precision of the results, so that they could be quickly resolved.

Laboratory Analysis Completed

EPA sampled over 1300 community water system wells and domestic drinking water wells nationally from April 1988 to February 1990. Laboratory analysis was completed in May 1990. EPA's Phase I Report on the Survey findings was released in Fall 1990. A Phase II Report, including relational analyses, is scheduled for release in Spring 1991.

Where to Go for More Information

This fact sheet is part of a series of NPS outreach materials, fact sheets and reports. The following additional fact sheets are available through EPA's Public Information Center (401 M Street SW, Washington DC 20460, 202-382-2080):

Project Summary	***Survey Design***	***Survey Analytes***
Glossary	***Summary Results***	
Fact Sheet for each detected analyte	***How EPA Will Use The NPS Results***	***Quality Assurance/ Quality Control***

Additional information on the Survey and on pesticides in general can be obtained from the following sources:

U.S. EPA Safe Drinking Water Hotline 1-800-426-4791 (In Washington, DC -- 382-5533) Monday-Friday, 8:30 am to 4:30 pm Eastern Time	Information on regulation of pesticides in drinking water
National Pesticide Telecommunications Network 1-800-858-7378 24 hours a day	Information on health effects and safe handling of pesticides
U.S. EPA Office of Pesticide Programs (OPP) Docket 401 M Street, SW Room NEG004 Washington, DC 20460 (202) 382-3587	Background documents for Survey (available for review)
National Technical Information Service (NTIS) 5285 Port Royal Road Springfield, VA 22161 (703) 487-4650	Copies of the NPS Phase I Report (available 1991) and NPS Phase II Report (when available)

If you are concerned about the presence of pesticides and nitrate in your private water well, contact your local or State health department. Other experts in your State environmental agency or agriculture and health department may also be helpful to you. If you receive your drinking water from a community water system and have questions about your water quality, contact your local community water system owner/operator or the State water supply agency.